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Passive Badge Assessment for Long-term, Low-level Air Monitoring on Submarines: Monoethanolamine Badge Validation

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14. ABSTRACT

Passive diffusion badges are being tested as a long-term, low-level method of analyte-specific air analysis onboard U.S. Navy (USN) nuclear submarines. Passive badge monitors for monoethanolamine (MEA) detection were tested. Long-term sampling efficiency was evaluated for a 28-day period by comparing the response of the passive badge to an active tube sampling method. Simultaneous exposure of badges and tubes, at concentration levels 100% and 20% of the U.S. Navy 90-day submarine-specific limits, was performed. High and low level concentrations were tested to examine the response range of the badge. Badge results were stable and reproducible at 100% of the USN 90-day limit (0.50 ppm) and were on average 25% lower than active sampling tube results. At the 20% level (0.10 ppm) the badge results were typically 14% lower than tube results.

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Passive Badge Assessment for Long-term, Low-level Air Monitoring on Submarines: Monoethanolamine Badge Validation

1.0 Introduction

The submarine is a unique working and living environment, as submariners are contained in this environment 24 hours a day for the duration of deployment. It is important to know and monitor the safety of the atmosphere to which they are exposed. Current methods of air monitoring onboard U.S. Navy (USN) nuclear submarines include the central atmosphere monitoring system (CAMS) and active, colorimetric tube sampling (Draeger). The CAMS provides continuous, real-time air analysis for only a few critical compounds. Draeger tubes provide real-time results for other species of interest, but sampling is not continuous. The Draeger tube methods are labor intensive and have poor reproducibility as the result of a manually operated hand pump, as well as multiple interpretations of the manually read tube results. This labor intensive method introduces the problem of human error and lack of reproducibility. Implementing passive badges would greatly reduce sources of error, as they are professionally analyzed and require very little human manipulation. They may supplement or even replace certain sampling procedures while providing continuous air sampling, relieving the sailors to perform other important duties onboard the ship. Additionally, numerous analytes can be tested at the same time using one or multiple badges.

For use on submarines, passive badges should provide continuous air monitoring for up to 28 consecutive days. Before the badges can be used in this application, they must be validated for long-term use, as they are currently only validated commercially for a normal 8-hour working day. To assess their long-term responses, for exposures up to 28 days, the badges were compared to commonly-used active sampling tubes. An exposure chamber was designed that would ensure that a homogeneous test vapor was delivered to both the tubes and the badges. Six of these chambers were manufactured to allow multiple concentration levels to be sampled simultaneously (1).

Monoethanolamine (MEA) is used on submarines to remove CO_2 from the air. It is, therefore, present and must be monitored in closed space environments. The OSHA exposure limit is 3 parts-per-million (ppm). Exposure to MEA may result in skin irritation causing redness, swelling, and lesions. Other symptoms of overexposure include apathy, poor appetite, and dermal effects ranging from ulceration to hair loss. However, because of the unique environment aboard submarines the USN 90-day limit for MEA is set at 0.5 ppm. Passive badge monitoring for MEA was evaluated for long-term exposures at 20% and 100% of the USN 90-day limit, 0.10 ppm and 0.50 ppm respectively. Lower levels were employed to assure that the 100% level could be accurately measured.

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2.0 Experimental

The test vapor was generated by infusing an MEA solution into a clean airstream using a programmable syringe pump (New Era Pump Systems #NE-1000). The MEA solution was made by diluting 99% MEA (Acros cat # 149580010) into deionized water, making 11700 µg/mL. MEA-specific badges were purchased from Assay Technology (Organic Amines #585). The fiberglass pad of the badge was coated with 1-Naphthyl isothiocyanate (NITC), which reacts with MEA to form a MEA-NITC derivative. The derivative was extracted into 5 mL of acetonitrile and allowed to desorb for at least 16 hours. The samples were then analyzed by HPLC (HP 1100), as indicated by Assay Technology.

The same chemistry was used by the active sampling tubes (SKC 226-30-18), which had NITC coated onto XAD-2 resin. The active tube samples were collected using a sample pump (SKC Airchek 224-PCXR7) to pull approximately 50 mL/min of vapor across each tube's substrate. The samples were analyzed by scoring and breaking open the tubes to transfer the resin into a clean sample vial. The sample was extracted and analyzed as described above for the badges. Results obtained from all samples were compared against a standardized curve covering the range of 1-100 µg/mL. The curve was generated by spiking tube and badge sampling substrates with increasing amounts of MEA and desorbing the samples in 5 mL of acetonitrile. An independent method of MEA capture and detection was used as a secondary verification of MEA exposure in the chamber.

2.1 Test Chambers

The test chambers were designed for the purpose of delivering a reproducible, homogenous test vapor, while simultaneously accommodating six passive badges and five active tubes. During this research, the badge design was modified by Assay Technology, resulting in reconfiguration of the badges in the chamber allowing six badges to be sampled instead of five. The chambers are comprised of multiple parts: introduction chamber, mixing baffles, badge plate, tube ports, and a fan, as shown in Figure 1. The chamber's body is tubular, chosen over a traditional rectangular shape to reduce "dead" air space within corners of the chamber. The body is 10.8 cm in diameter (ID) and 30.5 cm long. A plate within the chamber was reconfigured to hold six badges, each being exposed to a uniform airstream at a specified face velocity, as shown in Figure 2. The sampling rate of the MEA badge, as specified by the manufacturer, was 8.0 mL/min. To maintain this sampling rate, a minimal linear face velocity of $\geq$17 cm/sec, or 13 L/min, was sustained (2). The plate directed a total volume of 30 L/min of test vapor through the six 1.2 cm × 2 cm openings, one in front of each of the six badges, providing the appropriate face velocity. The fan at the back of the chamber pulled the test vapor through the chamber as it was introduced, at approximately 29 L/min. A slight overpressure in the chamber prevented room air from leaking into the system. Two baffles at the front of the chamber aided in mixing the vapor stream.

2.2 Experimental Design

Clean, dry air was obtained by passing compressed house-air through dual-tower molecular sieves, to remove moisture and CO_2, then distributed between five mass flow controllers, each set to deliver approximately 30 L//min of the air to its respective test chamber. The MEA vapor was generated by infusing an aqueous MEA solution into the clean, dry airstream. A programmable syringe pump dispensed the MEA solution at a constant rate of 30 µL/min, providing a vapor concentration of 4.7 ppm, for the duration of the pulsed exposure. The MEA/water solution also humidified the air as the vapor was formed. Deionized water was infused into clean air for the control chamber. The flow rates of all controlled airstreams were measured using a Dry-Cal flow meter.

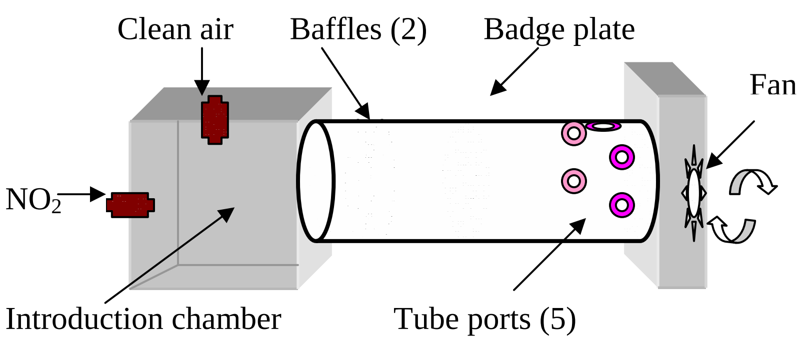


Figure 1. Diagram of a validation chamber.

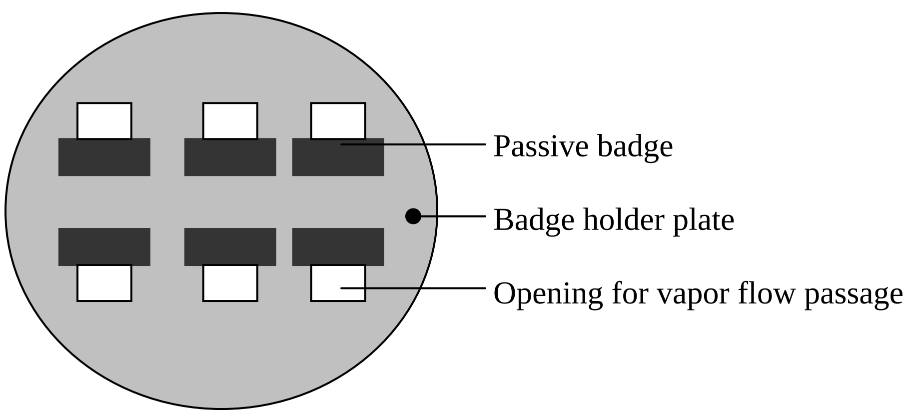


Figure 2. The badge plate, with 6-badge capacity.

The badges were inserted into the badge plate, all badge faces facing the opening above it. The active sampling tubes were connected to adjustable, low-flow 4-tube manifolds, SKC 224-26-04. Each chamber's manifold allowed a single pump to sample for the four tubes attached. The pumps were set to pull 200 mL/min, to be distributed among the four sampling tubes providing a nominal sampling rate of 50 mL/min per tube. Due to slight differences in the tubes as a result of manufacturing processes, the pressure drop across the tubes varied resulting in small variations of flow though the tube. The flow rate of each tube was measured independently using an in-line Sierra mass flow meter

before being inserted into the chamber and again before its removal. The average flow rate, per tube, was used when analyzing the final data results.

The analyte exposures were generated using the "pulse" method. Instead of exposing the samples to the analyte vapor continuously, the exposures were delivered only three times per week. The concentration of the pulsed vapor was the same in all chambers, 4.7 ppm, however the length of the pulse was different for the 20% versus 100% level chambers. Each pulsed exposure lasted 72 minutes for the 20% level and 360 minutes for the 100% level. The cumulative, time-weighted-average (TWA) exposure per week was equivalent to a continuous exposure at the 20% and 100% levels. Clean air was passed through the chambers continuously when the analyte was not being delivered. Running the pulse method was advantageous in monitoring system mechanics to ensure that all of the equipment was functioning properly. It may also be a more realistic demonstration of how the badge might respond to an instantaneous toxic level exposure to a hazardous compound.

The experiment ran for 4 weeks (28 days). Chambers "A" and "B" tested the 20% level, and Chambers "C" and "D" tested the 100% level. To monitor the progress of the experiment, a scheduled number of badges and tubes were systematically removed per week. These badges and tubes were analyzed to guarantee that the system was functioning properly and to assess the behavior of the badges over time. The data was catalogued each week and used to compile a final data analysis at the end of the 28-day testing period. The schedule is illustrated in Figure 3. Each week three badges were removed from a low-level testing chamber and three badges were removed from a high-level testing chamber. Badges the first week were removed from chambers A and C. The next week, badges were removed from chambers B and D. This pattern was repeated for the duration of the validation. Simultaneously, two tubes were removed from each chamber following the same procedure as for badges. New badges and tubes were inserted in the chambers in place of the removed samples. At the end of the 28 days all of the remaining tubes and badges were removed from the chambers. Collectively, the data were representative of the first 7, 14, 21, and 28 days and for the last 21, 14, and 7 days. The total numbers of data points were as follows:

7 days	20 data points	
14 days	20 data points	Total = 70 data points
21 days	20 data points	
28 days	10 data points	

2.3 Independent Method

The vapor concentration within the chamber was verified for each pulse using an independent method developed and validated at NRL (3). MEA vapor was collected onto silica gel tubes (SKC #226-22) for the duration of the pulsed exposure using SKC pocket pumps 210-1002, set at a sampling rate of 50 mL/min. After the pulse, the exposed tubes were removed from the chamber and analyzed, while new silica gel tubes were inserted into the chamber to monitor the clean air between the consecutive pulses. This scheme monitored for reverse diffusion of the analyte into the air or additional accumulation of

analyte from adjacent surfaces. The clean-air silica gel tubes were removed prior to the beginning of the next pulse. After extraction and derivatization, the samples were analyzed by GC/MS (Varian Saturn 4D), using chemical ionization detection, then used to compile a final data analysis at the end of the testing period. Results of the exposed and clean tubes were added together to obtain a sum of TWA MEA exposures.

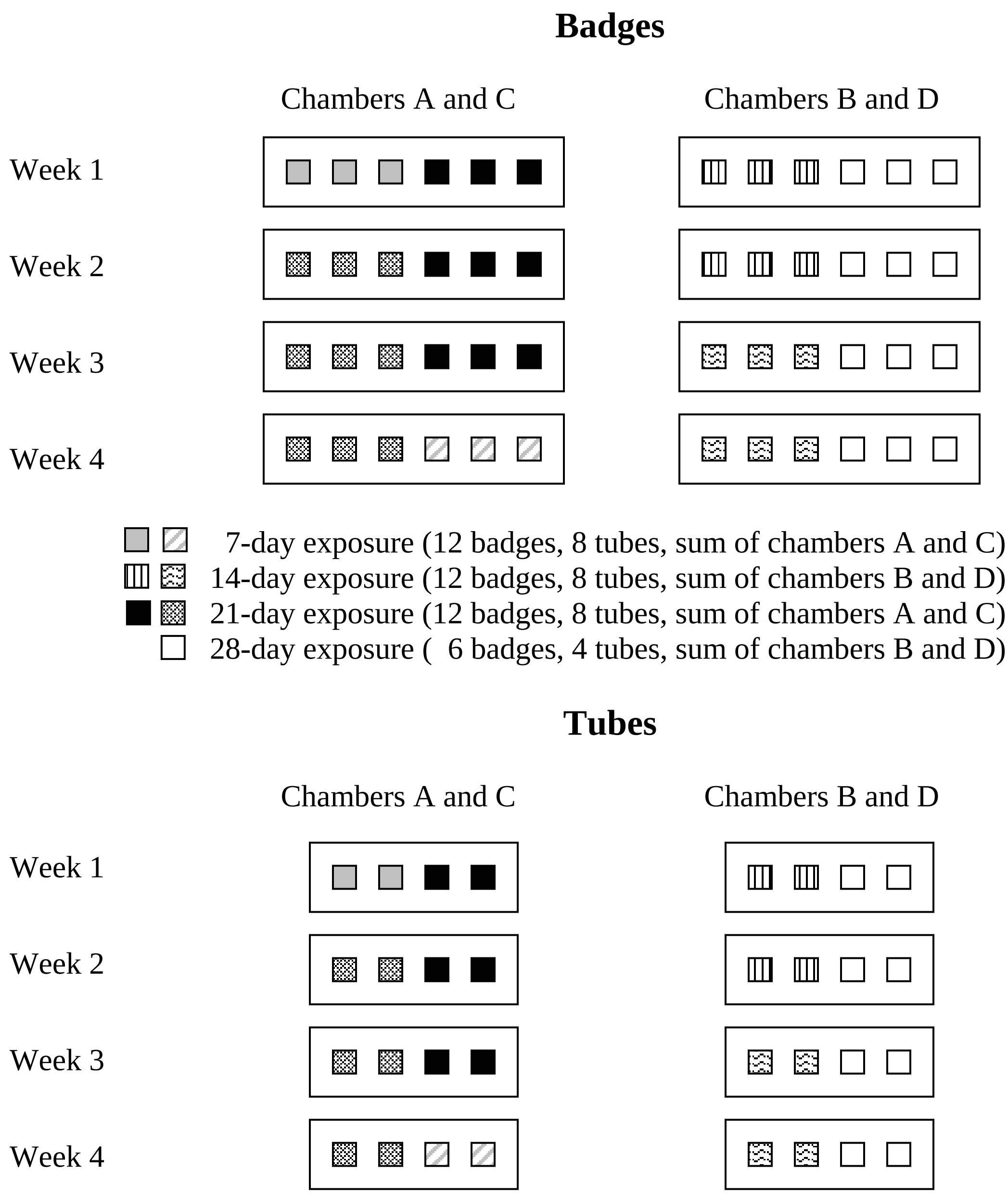


Figure 3. Schedule of badge and tube removal/replacement. Overlapping series helped average-out any errors in MEA concentration over the long exposure.

2.4 Analysis

Each week, following removal from the test chamber, the tubes and badges were extracted for MEA analysis as indicated by Assay Technology. Analysis was similar to

OSHA PV2111 analytical method (4). The faces of the badges were opened to remove the fiberglass sample pad. The pad was transferred to a clean sample vial filled with 5 mL of acetonitrile solvent. The glass sample tubes were scored then broken open to empty the contents into a clean sample vial filled with 5 mL of acetonitrile solvent. The tube and badge samples were vortexed for 1 minute then set aside to be analyzed by HPLC the following day to ensure complete desorbtion of the analyte from the sorbent. The reaction of MEA with NITC is a slow process and requires more than 8 hours to proceed to completion, see Figure 4 for the chemistry of the derivatization reaction. Specifications of the HPLC included: a Restek Ultra C18, 5 µm, 150 x 4.6 mm reversed phase column, water/acetonitrile mobile phase (Table 1), and a 20 µL sample loop injection. The method runtime was 7 minutes with an elution rate of 1.5 mL/min. The retention time of the MEA-NITC derivative was approximately 1.4 minutes and the excess NITC at 6.1 minutes. The spectra were read at 365 nm. Sample data obtained from the HPLC was compared against the standard curve. The range of the curve was 1-100 µg/mL. Dilutions were made for samples with concentrations outside of the range of the curve.

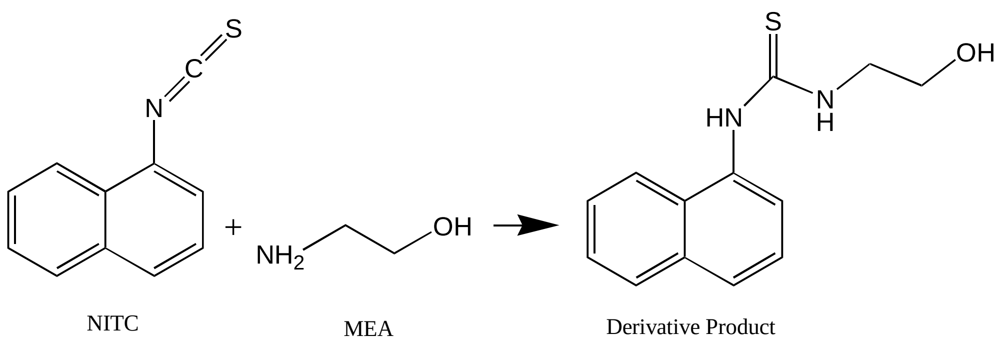


Figure 4. Chemistry of the sampling media.

Table 1. Timetable of events for the HPLC mobile phase.

Minute	Water, %	Acetonitrile, %
0.0	50	50
3.0	50	50
4.0	0	100
7.0	0	100

3.0 Results and Discussion

Data was gathered and compiled on a weekly basis by removing a scheduled number of tubes and badges from each chamber. The raw data are given in Tables 2 and 3. Calculations were based on weekly measurements of the gas analyte, airstreams, and sampling rates. The sampling rate of the badges was assumed to be constant for each badge, whereas the sampling rate of each tube varied slightly. The flow rates of the tubes were measured upon introduction to the system and again prior to the tube's removal

from the chamber. The average flow measurement, per tube, was used when calculating the concentration

Table 2. Raw data for the active sampling tubes.

20% Level, 0.10 ppm					100% Level, 0.50 ppm				
Days of exposure	Total µg sampled	Sampling rate, L/min	Conc in chamber, ppm	%RSD	Days of exposure	Total µg sampled	Sampling rate, L/min	Conc in chamber, ppm	%RSD
7	139.14	0.0505	0.109		7	643.03	0.0510	0.501	
	131.22	0.0480	0.109	0.55		682.33	0.0510	0.532	4.19
14	309.14	0.0510	0.120		14	1077.42	0.0500	0.428	
	293.73	0.0540	0.108	7.65		1062.18	0.0510	0.414	2.41
21	280.07	0.0495	0.075		21	1799.25	0.0495	0.481	
	318.71	0.0530	0.080	4.31		1873.67	0.0495	0.501	2.87
28	826.07	0.0535	0.153		28	2762.93	0.0500	0.549	
	715.83	0.0535	0.133	10.11		2897.25	0.0495	0.581	4.07
21	364.83	0.0560	0.086		21	2303.94	0.0470	0.649	
	362.62	0.0510	0.094	6.18		2318.98	0.0480	0.640	1.03
14	252.02	0.0560	0.089		14	1651.56	0.0520	0.631	
	260.25	0.0550	0.094	3.55		1395.18	0.0510	0.543	10.54
7	99.60	0.0510	0.078		7	930.57	0.0485	0.762	
	119.02	0.0540	0.088	8.54		648.44	0.0495	0.520	26.66
		average ppm		0.10			average ppm		0.55
		average %RSD		5.84			average %RSD		7.39

Table 3. Raw data for the passive badges.

20% Level, 0.10 ppm				100% Level, 0.50 ppm			
Days of exposure	Total µg sampled	Conc in chamber, ppm	%RSD	Days of exposure	Total µg sampled	Conc in chamber, ppm	%RSD
	16.27	0.081			88.77	0.441	
7	11.73	0.058	17.42	7	72.12	0.358	11.70
	12.80	0.064			73.85	0.367	
	31.72	0.079			147.75	0.367	
14	33.00	0.082	8.96	14	173.57	0.431	10.09
	37.53	0.093			145.20	0.361	
	53.86	0.089			243.25	0.403	
21	31.63	0.052	27.68	21	257.53	0.426	4.75
	38.24	0.063			267.44	0.443	
	77.58	0.096			350.79	0.436	
28	69.05	0.086	6.19	28	391.77	0.486	5.52
	70.89	0.088			372.69	0.463	
	58.82	0.097			257.74	0.427	
21	26.79	0.044	40.38	21	240.60	0.398	4.35
	36.44	0.060			261.13	0.432	
	38.23	0.095			184.00	0.457	
14	37.60	0.093	8.55	14	170.73	0.424	3.83
	43.80	0.109			175.09	0.435	
	18.71	0.093			88.12	0.438	
7	18.62	0.092	9.13	7	84.81	0.421	6.08
	21.78	0.108			78.13	0.388	
	average ppm		0.08		average ppm		0.42
	average %RSD		16.90		average %RSD		6.62

accumulated by each tube. All sample values, tubes and badges, were calculated to reflect the concentration within the chamber, respective to each sample. With all data presented in the same manner, direct comparisons could be made. Data from the control "clean" chamber showed no indication of MEA contamination, indicating that there were no interferences causing false-positive results. Accumulation of the analyte onto badges

was consistently lower than accumulation onto tubes. The relative standard deviation (%RSD) of tubes per week ranged from 1.0 – 26.7%, with an average of 7.7%. The %RSD of badges per week ranged from 2.6 – 40.4%, with an average of 12.3%. When comparing the results of badges and tubes of the same exposure period, the %RSDs ranged from 7.7-30.2%, with an average of 19.7% at the low concentration level (20%), and ranged from 8.9-29.9%, with an average of 17.6% at the high concentration level (100%). Results collected weekly for tubes and badges were consistent, as indicated by the low RSD values. However, the increased RSD levels, when comparing badges to tubes, verify that the badge results were different than the tube results, Table 4. On average, the badge results were 20% lower than that of the tubes.

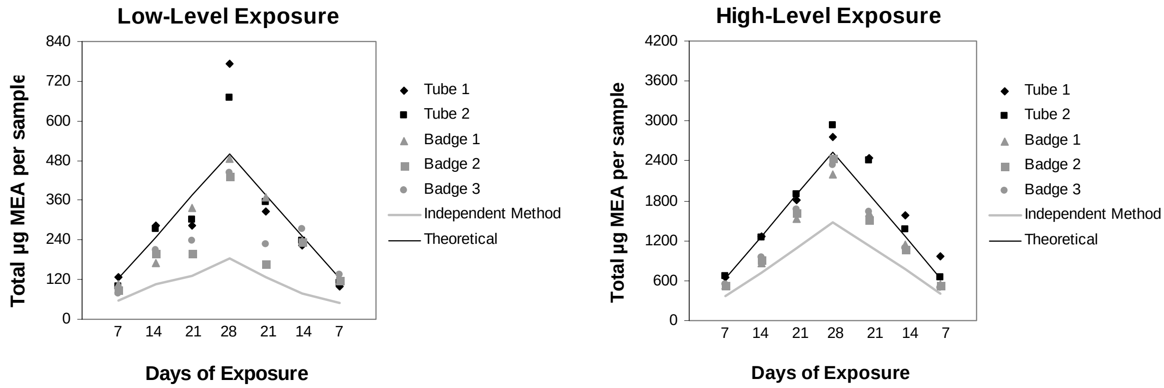
Table 4. Weekly comparison of tubes and badges. Boxes 1, 2, 3, and 4 are the same as Chambers A, B, C, and D, respectively.

20% Level									
Box 1		Tube Conc †	Badge Conc †	%RSD	**Box 2**		Tube Conc †	Badge Conc †	%RSD
	1	25.72	21.53			1	57.04	34.24	
7 days	2	20.18	17.99	**18.8**	14 days	2	54.59	39.71	**21.8**
	3	NA	15.66			3	NA	41.51	
	4	56.58	67.33			4	154.40	96.98	
21 days	5	60.13	39.54	**19.9**	28 days	5	133.80	86.31	**27.2**
	6	NA	47.81			6	NA	88.61	
	1b	65.15	73.52			1b	45.00	47.79	
21 days	2b	71.10	33.49	**30.2**	14 days	2b	47.32	47.00	**7.7**
	3b	NA	45.56			3b	NA	54.75	
	4b	19.53	23.39						
7 days	5b	22.04	23.27	**12.1**			average %RSD		19.7
	6b	NA	27.23						
100% Level									
Box 3		Tube Conc †	Badge Conc †	%RSD	**Box 4**		Tube Conc †	Badge Conc †	%RSD
	1	131.31	104.82			1	251.87	172.11	
7 days	2	133.81	106.11	**12.1**	14 days	2	248.87	184.41	**18.1**
	3	NA	110.12			3	NA	190.71	
	4	363.48	304.06			4	552.59	438.49	
21 days	5	378.52	321.91	**8.9**	28 days	5	585.30	489.72	**12.0**
	6	NA	334.29			6	NA	465.87	
	1b	490.20	322.18			1b	317.61	230.01	
21 days	2b	483.13	300.75	**24.4**	14 days	2b	273.57	213.41	**17.7**
	3b	NA	326.41			3b	NA	218.86	
	4b	191.87	110.15						
7 days	5b	131.00	106.01	**29.9**			average %RSD		17.6
	6b	NA	97.66						

† Concentrations are µg/mL and represent a sample flow rate of 50 ccpm.

Although the badges did not provide the same level of response as the tubes, the badges did have the same behavioral patterns as the tubes, Figure 6. The patterns of behavior were also verified by comparison to the independent method. This indicates that the badges perform in a way that is predictable. The low response of the badges may be attributed to the diffusion rate of the badge. It is likely that the sampling rate of the badge was different in our experimental setup than that suggested by the manufacturer. The performance of all samples appeared to be superior at the higher concentration level and closer to linearity. The independent method showed about a 50% recovery of the MEA, which cannot be readily explained. Perhaps something in the extraction procedure was changed unintentionally, resulting in loss of recovery. However, the response of the

independent method was consistent and highly reproducible at the high concentration level, Table 5.



(a) (b)

Figure 6. Accumulation of MEA onto tubes and badges for 28 days at (a) 20% and (b) 100% of the 90-day limit.

Table 5. Raw data for the independent method of MEA verification. Boxes 1, 2, 3, and 4 are the same as Chambers A, B, C, and D, respectively.

Independent Method						(28 days)
		Week 1†	Week 2†	Week 3†	Week 4†	SUM
20% Level	Box 1	9.34	10.85	3.85	9.90	33.94
	Box 2	13.02	7.75	6.46	10.31	37.55
	average	11.18	9.30	5.16	10.11	35.74
	%RSD	23.3	23.5	35.8	2.9	7.2
100% Level	Box 3	74.12	60.52	72.17	80.53	287.34
	Box 4	72.70	67.62	73.84	79.35	293.51
	average	73.41	64.07	73.01	79.94	290.43
	%RSD	1.4	7.8	1.6	1.0	1.5

† Sum of 3 pulses per week, µg/mL

4.0 Conclusions

The results provided by the four sampling chambers were compared to establish response patterns of the passive badges, relative to active tubes, to MEA over a 28-day exposure. The badges and tubes continued to accumulate the analyte for 28 days, with accumulation of the analyte onto badges consistently about 20% lower than accumulation onto tubes. Reproducibility among passive badges and active sampling tubes was most successful at 100% of the USN 90-day limit, 0.50 ppm. The average RSD of badges at 0.50 ppm was <10%, indicating that the results were reproducible and predictable. Further studies may show that the sampling rate of the MEA-specific badge is different in our setup than that of the manufacturer. Since the badge results are stable and reproducible at the 100%

level, a correction factor may be implemented for a more accurate result until the sampling rate can be reassessed. The passive badges should provide a reliable result for long-term identification of MEA at, or near, 0.50 ppm following proper validation of the MEA badge sampling rate. Monoethanolamine can be detected at 20% of the USN 90-day limit with the use of passive badges. However, results provided by the badge may not be accurate until more work is done.

5.0 References

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